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SCIENCE DIRECT
Industrial Crops and Products 23 (2006) 264–272
INDUSTRIAL CROPS AND PRODUCTS
AN INTERNATIONAL JOURNAL
www.elsevier.com/locate/indcrop

Separation of particles from ethanol/maize extracts: An inexpensive alternative to centrifugation☆

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Received 14 January 2005; accepted 16 August 2005

Abstract

An important aspect of extraction using an organic solvent that is often ignored in many laboratory scale studies is thorough solvent recovery. Although most of the solvent can be recovered with a centrifuge, the solvent left on the 'dry' stream must be evaporated. A custom built pilot-scale settling tank was used to separate maize particles from ethanol extracts into water with little dilution of the extract liquid. The larger particles that settled in the first one-fourth of the tank were carried out by a continuous water flow 76 cm below the extract layer. The large particles had 80% higher protein mass fraction than the smaller particles that collected in the bottom of the settling tank downstream from the extract inlet. Water flow was confined to the bottom of the tank and extracted particles were prevented from accumulating in the settling tank with a much lower water/extract flow rate ratio than needed for a smaller settling tank. The mass ratio of entrained extract liquid/settled solids (0.5) was one-half that observed in previous methods using smaller tanks. This is caused by a more stable extract/water interface. The yield from finer, 1 mm meal, was slightly lower than 2 mm meal and increased extract liquid entrainment. Consequently, the 2 mm particle size is the minimum that should be used with this process.

Keywords: Particle separation; Maize; Extraction; Ethanol; Protein; Zein isolate

1. Introduction

An inexpensive process to isolate zein (ethanol-soluble maize protein) is needed. Maize meal has been extracted using extract centrifugation (Dickey et al., 2002). The technology developed for maize could be used to extract protein from other cereal grains (Erasmus and Taylor, 2003). Centrifugation of ethanol extracts was not satisfactory because evaporating the ethanol from the solid product stream, which contains 45% extract liquid is expensive. Thus, cost considerations required a separation method that would allow recovery of more extract liquid from each extraction batch without evaporation. It was determined that separation by a settling process is feasible when both the particles and liquid are too valuable to be discarded and where drying of the settled particles for subsequent use is unnecessary. The separated starch particles in the feedstock will be converted in an aqueous suspension and then fermented. Adequate settling requires minimizing entrainment of the extract liquid by the settling particles and continuous settling

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doi:10.1016/j.indcrop.2005.08.006

must remove settled particles from the water layer at their settling rate.

Settling extract particles into water was investigated using a 35-l tank (Dickey et al., 2003). For the particles to be removed by water flow at the bottom of the tank, the water flow rate must be high enough to move the settled particles to the tank outlet, but not high enough to cause convective mixing between the water and the extract layer at the top of the tank. Tests showed that a flat base must be inclined at least 35° from the horizontal toward the outlet with water flowing along the base to keep particles from accumulating (Dickey et al., 2003). Furthermore, the particle settling must be fast enough that removal can be accomplished within a given size tank. This was achieved by keeping the extract layer thickness around 5 cm. Based on results from earlier tests, replacing centrifuges with settling tanks reduced the estimated capital cost of zein recovery by 50%. The mass of extract liquid entrained by the settled particles was found to be about equal to the mass of the particles, i.e., the mass ratio, L/S, was about 1.

The objective of this work was to expand the small-sized experiments to evaluate the effect of scaling on the entrainment. Less entrainment was expected if the water flow could be kept at the bottom of the tank, further from the extract/water interface than possible in a smaller tank. It was also possible with the larger tank to measure the vertical ethanol concentration profile in the water below the extract.

2. Experiment

2.1. Extraction of maize meal

Two types of experimental process were used. Shelled maize was obtained from a local feed mill. In the first experiment, it was cleaned by manual removal of cob and stalk pieces and then milled (Wiley, Model 1, Swedesboro, NJ) with a 2 mm screen. In the second, the maize was cleaned with a dockage tester (Carter Day, International, Minneapolis, MN) and a mini-aspirator (Kice Industries, Wichita, KS) and milled with a 1 mm screen. The milling for each run was made on the 2 days prior to extraction and settling. Subsequent to these runs, a batch of shelled maize was cleaned with the dockage tester and mini-aspirator and divided into two 11.2 kg portions. One portion was milled with the 2 mm screen (11.1 kg recovery) and one with the 1 mm screen (10.7 kg recovery). The meal from each portion was then sized on a testing sieve shaker (RoTap, W.S. Tyler Co., Cleveland, OH). One kilogram of meal was shaken for 30 min on a stack of sieves (U.S. Standard screens 16, 20, 25, 28, 40 and 60). At the end of the period, the different-size particles that had been passed through were collected in seven separate bags. The particles stuck in the screens were added to the unsieved powder from which another 1 kg was taken, shaken, and collected. This procedure was continued until all of the powder was sieved. The mass fraction for each size interval is shown in Table 1. Corn milled with the 1 mm screen in the mill had equal or more oil in particles smaller than 0.84 mm and less in the larger particles (Dickey et al., 1997). The minimum in mass fraction for the interval 0.71–0.59 mm was also evident in the earlier examination. It seems that the finer milling and following meal cleaning removes more starch containing particles than the coarser milling.

The meal was mixed in a jacketed tank for 90 min at 50 °C with 70% ethanol solution or extract liquid recovered from previous runs and brought up to 70% ethanol by adding the necessary amount of ethanol. The meal slurry was agitated by two scraper blades attached to a central shaft rotating at 10 rpm and circulated out of the tank with a centrifugal pump (Fristram, model FP702, Middletown, WI) at 40 kg/min and back into the tank to increase the disruption of the corn particles.

2.2. Chemical analysis

With the exception of the solid samples, all analyses were performed on the upper soluble liquid layers of samples. All analyses were done in duplicate. Oil content was determined by hexane extraction in a separatory funnel. The upper hexane layer was collected into tared beakers and oil determined gravimetrically.

The oil content of the solid samples was determined by hexane extraction (Moreau et al., 2003). A known volume of liquid sample was dried under a stream of nitrogen, weighed, and the solid material collected for pyrolysis to determine protein content. The standard pyrolysis procedure was used (AOAC, 1998; AACC, 1995). Moisture content of the solid samples was determined using AACC Method 44-19. Starch was determined using an enzymatic assay with the AOAC Method 46-30 and AACC Method 32-32. The composition of the milled maize is shown in Table 1.

2.3. Settling of extract

A 830-l, stainless steel, settling tank with vertical triangular end walls with 2.44 m × 1.12 m rectangular sides was built (Fig. 1). One side had three 0.91 m × 0.20 m windows and the 2.44 m × 1.22 m lid had two 0.91 m × 0.28 m windows and one 1.14 m × 0.28 m window with a downward-facing light

Table 1
Solids, protein, oil, and starch content of 2-mm and 1-mm fractions of milled maize

Particle size (mm)	Meal size	Mass fraction	Solid (%)	Protein (%)	Oil (%)	Starch (%)
>1.168	2 mm	0.433	87.4	6.34	1.67	47.2
	1 mm	0.002	90.8	6.31	3.07	65.0
1.168–0.850	2 mm	0.236	87.6	9.68	3.18	53.9
	1 mm	0.559	90.2	8.67	2.75	64.7
0.85–0.710	2 mm	0.101	88.7	7.55	3.75	56.5
	1 mm	0.274	89.6	7.32	3.56	60.6
0.710–0.590	2 mm	0.025	88.2	6.49	3.65	63.2
	1 mm	0.024	89.8	9.52	3.49	66.4
0.590–0.420	2 mm	0.082	88.5	7.10	3.79	58.4
	1 mm	0.122	89.7	8.10	4.12	60.6
0.420–0.250	2 mm	0.084	87.8	5.72	3.12	69.3
	1 mm	0.013	89.8	10.51	3.96	62.6
<0.250	2 mm	0.039	88.3	5.67	3.01	65.8
	1 mm	0.006	89.9	6.09	4.01	68.1
Calculated average[a]	2 mm		87.8	7.24	2.63	52.3
	1 mm		89.9	8.25	3.18	61.9
Milled[b]	2 mm	1.0	87.3	6.47	2.51	53.6
	1 mm	1.0	90.0	10.54	3.11	63.1
Whole kernel		1.0	86.1	7.81	2.64	60.8

[a] The sum of the products of mass fraction (columns 3) and composition percentage (columns 4,5,6, or 7).
[b] Sample taken from milled batch before sifting.

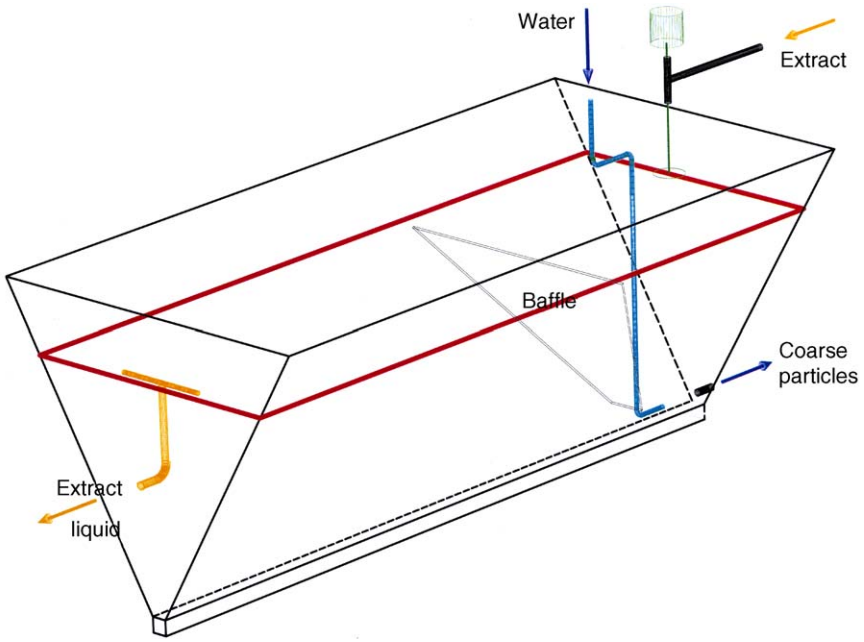

Fig. 1. The 830-l, V-cross section settling tank, with flow direction of the extract and coarse particle transporting stream.

mounted over it. The tank had a lid attached to a lever that allowed it to be lifted with an air cylinder. A rubber, deformable tube type gasket was attached to the top of tank, sealing it when the lid was lowered. A triangular baffle extended across the tank, just below the interface dividing the water layer into two chambers. The baffle was oriented about 60° from the horizontal so that particles descending on it could slide downward toward the water outlet at the bottom of the tank. Thus, particles settling from the first 0.60 m of the extract layer were collected at the space between the water inlet tubing and the outlet in the (extract feed) end wall of the tank.

The extract was pumped into the settling tank (A in Fig. 2) using a Masterflex L/S pump (Cole-Parmer Instrument Co., Vernon Hills, IL). The extract was conveyed at 1.32 kg/min through a size-36 Tygon® tubing to the middle arm of a tee mounted in the settling tank lid, 7 cm from one end. An agitator shaft extending 16.5 cm below the lid was mounted through the upper arm of the tee. A 11.4-cm diameter disk rotating at the bottom of the shaft deflected extract flowing down the shaft outward to distribute the incoming extract across the top of the extract liquid layer. When all of the extract that could be pumped from the extraction tank had been removed, the pump was turned off. The valves on the extraction tank and on the inlet of the settling tank were closed. Residual extract consisting mostly of particles left in the extraction tank was rinsed out with a known amount of water, weighed, and the ethanol content of the liquid phase measured. This allowed determination of the mass and solid content of the material left in the extraction tank.

Settled extract (liquid) overflowed through a 27-cm long slot in the top of a 2.5 cm diameter tee whose ends were capped and whose middle arm is aligned vertically downward (D in Fig. 2), thereby holding the extract liquid/air interface 23 cm below the top of the tank. The middle arm of the tee was attached to a descending tube connected to a port in the tank through which the extract liquid flowed to a collection tank (Tank 1 in Fig. 2). Extract liquid can be fed to the settling tank and drain through the outlet port at rates up to1.6 kg/min without causing the height of the air/liquid interface to rise.

A problem became apparent when an ethanol layer of uniform thickness was formed to cover the water layer. When the extract was pumped in from the feed end, it would form a thick layer on that end while water overflowed through the outlet on the other end of the tank. Eventually, the extract layer would extend to the outlet, but by then, a substantial amount of the extract would have been consumed and diluted. To overcome this startup problem, about 341 (1.5 cm at interface) of 45–60% ethanol was slowly pumped into the tank through the slotted tee outlet (D′ in Fig. 2), onto the water that was a few cm below the slot. The ethanol solution spread across the top of the water in the settling tank, establishing a fairly uniform layer. After the buffering solution layer was in place, water was pumped into the bottom of the settling tank (through valve V2 in Fig. 2) until the liquid level was just below the overflow slot. The water inlet and outlet on the other end of the settling tank were 76 cm below the overflow slot.

Settled particles descending on the feed end of the settling tank were carried out with a water stream at the bottom of the tank. In-flowing water was pumped through tubing penetrating the lid extending to the bottom of the tank. Water was pumped out (Fristram, model FP702, Middletown, WI) at 40 kg/min through an outlet port (D″ in Fig. 2) in the center of the end wall.

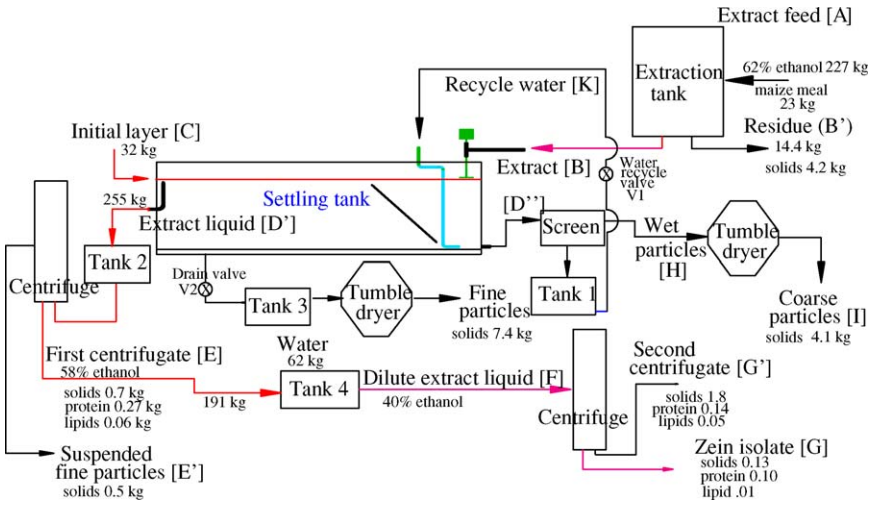

Fig. 2. Schematic drawing of settling and particle drying of 2 mm maize meal.

The water stream leaving the bottom of the extract feed end of the settling tank flowed to a 230T NCH mesh (74-μm open area) sieve (Sweco, vibro-energy separator, Florence, KY) that removed most of the larger particles. The particles collected in clumps that rolled out of the sieve and were collected in pails at a solids content of 25–30% and were later dried in a double cone tumble dryer (Patterson-Kelley, East Stroudsburg, PA) (between streams H and I in Fig. 2). The liquid passing through the sieve drained into a 60-l tank (Tank 1 in Fig. 2) from where it was pumped with a hose pump (Cole-Parmer Instrument Co., Model B/T variable speed, Vernon Hills, IL) back to the settling tank. The water level in the settling tank was kept steady by manually adjusting a valve in the water return line (water recycle stream K in Fig. 2).

Finer, more slowly settling particles descended to the bottom of the settling tank downstream from the extract feed end. These particles were too small to sieve from the water at practical rates and collected in the bottom of the settling tank. They were removed by pumping after the extract pumping stopped.

The extract liquid that drained from the settling tank was collected in the 60-l tank HDPE (high density polyethylene) (Tank 2 in Fig. 2). Liquid was pumped with a dual-headed tubing pump (Cole-Parmer Instrument Co., Model 77601-10 I/P variable speed, Vernon Hills, IL) from this tank to an in-line coriolis sensor (MicroMotion, model CM050, Boulder, CO) to measure the fluid density, flow rate, and temperature followed by a Model 8100 in-line ultrasonic probe (Rhosonics, Baarn, Netherlands) to determine the ethanol content. The probe was mounted with the long axis vertical to insure that solids would not accumulate in it. The ultrasonic probe was calibrated in two ranges of 70–50% and 0–10% ethanol. Probe accuracy was a few percentage points beyond the range. Ethanol content between 40 and 15% ethanol was determined by specific gravity. Extract liquid leaving the ultrasonic probe initially flowed to a 123-l HDPE tank. The ethanol concentration of the settled extract liquid indicated by the ultrasonic probe was monitored and when the value reached 55%, and was judged to have reached a steady composition, the extract liquid stream was collected in the 246-l tank (VT0065-23, Den Hartog Industries, Hospers, IA) that sat on an electronic floor scale (Champ II with CD-11 readout, Ohaus, Pine Brook, NJ). These tanks are not shown in Fig. 2, which represents the extract batch separation carried out over several days.

Samples of extract liquid flowing to the collection tanks were taken during the settling and analyzed later for ethanol, oil, protein, and solid content.

In the first run, after all of the extract that could be pumped from the extraction tank had been removed, the residual extract in the tank was rinsed out with 50 kg of water, then mixed thoroughly and allowed to settle. The specific gravity of the liquid phase was then measured using a hydrometer. The specific gravity was used to calculate the ethanol content of the (extract) liquid left in the extraction tank. The clear liquid was carefully decanted from the settled dilute residual extract and the damp particles dried in a 140-l vacuum tumble dryer. Samples of the extract liquid product were taken and analyzed later for ethanol, oil, protein, and solid content.

After the extract layer had been removed from the settling tank, the fine particles and remaining liquid in the downstream section of the settling tank were pumped

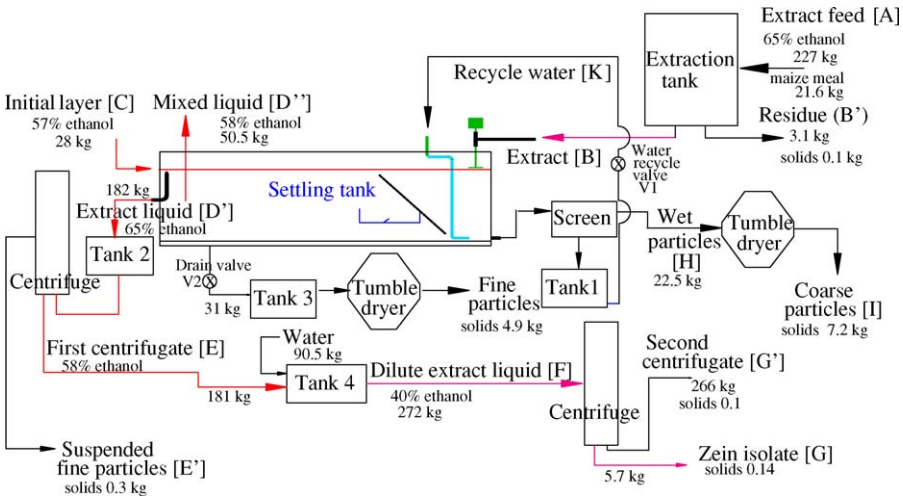

Fig. 3. Schematic drawing of settling and particle drying of 1 mm maize meal.

through Drain valve 2 in Fig. 2 into a 123-l HDPE tank (Tank 3 in Fig. 2) from which the liquid was drained. The wet particles were dried in the tumble dryer.

In the second run (Fig. 3), after all of the extract liquid product (stream D′ in Fig. 3) was drained through the slotted tee outlet (without raising the water level), 495 kg of liquid was slowly pumped from the bottom of the settling tank through drain valve (V2 in Fig. 3) which lowered the liquid level to 49.5 cm. The specific gravity of the pumped liquid indicated an average ethanol concentration of 0.5%. The remaining liquid (stream D″ in Fig. 3) was pumped from the top of the liquid through an outlet tube extending vertically through a port in the tank lid to the top of the liquid. This tube was inserted after the level was pumped down to 49.5 cm. The liquid was pumped through the ultrasonic and coriolis sensors and was collected in a 246-l tank (not shown in Fig. 3); thus, the ethanol content and the weight of the outflow could be continuously monitored. The presence of bubbles in the outflow indicated when the liquid being pumped out had dropped to the end of the tube and the outlet tube was pushed down through the port fitting 0.635 cm. This process was continued for 66 increments until the particles at the bottom of the tank were reached. The results of this process are shown in Fig. 4. The concentration trend was converted to a profile (in height of the settling tank contents prior to draining) using an equation based on tank volume measurements:

$$V = 0.150H^2 + 0.492\ H + 1.14$$

where V is the liquid volume (l) in the tank, at liquid height H (cm) from the bottom of the tank. This relation of V and H was necessary because the tank bulged as it was being filled. The tank walls were thin (3 mm). As with the first run, the fine particles, and the liquid

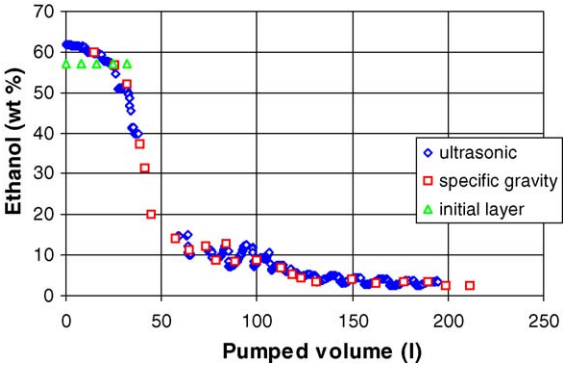

Fig. 4. Final extract liquid/water mixture profile in the settling tank. Initial layer (△) made from pure ethanol and water. Ethanol concentration of mixture measured by ultrasonic sensor (◊) or specific gravity (□).

remaining at the bottom of the tank were pumped into a 123-l HDPE tank (Tank 3 in Fig. 3) and the mixture was allowed to settle for 3 days. Then, the upper layer of liquid was drained and the remaining slurry of fine particles was dried in the tumble dryer.

2.4. Dilution and centrifugation of extract liquid

Fine particles that remained suspended in the settled extract liquid were separated with a AS26 Sharples® Super-Centrifuge (Alfa Laval Separation Inc., Warminster, PA). The centrifugate from this centrifugation step was diluted with water to 40% ethanol to precipitate the zein. The dilute extract liquid was centrifuged after setting overnight. The liquid was pumped to the centrifuge at 960 ml/min.

3. Results

Compositions of samples of various streams are listed in Table 2. Masses for product and intermediate streams for the two runs are shown in Figs. 2 and 3. In the first run, 23 kg of corn meal was extracted and 235.6 kg of extract was settled in 37 min. No interfacial layer of particles was evident in the settling tank. The ethanol recovered in the 255 kg of extract liquid drained from the tank at 58.9% ethanol corresponds to 242 kg of extract liquid at the concentration fed to the settling tank, 62%. As shown in Table 2, 4.1 kg of particles was left in the extraction tank.

In the second run (Fig. 3), 21.6 kg of 1 mm corn meal were extracted and 249 kg of extract settled in 55 min. Masses for product and intermediate streams for this run are shown in this figure. While settling the extract, an initial 50.5 kg of product liquid at 62% ethanol was recovered, followed by 147.5 kg at 65%. The average ethanol content of the 198 kg was 64%. One reason for draining a portion of the water in the settling tank, after settling, and then pumping out the remaining liquid from the top, was to measure the ethanol content at the top of the water layer and to determine if a visible oil layer would be created after the 495 kg of water was drained from the bottom of the settling tank. By slowly draining the triangular cross section tank from the bottom, the liquid in the upper layers collected into thicker layers. By measuring the ethanol by removing the liquid from the top in small height increments, the determination of the ethanol concentration in a given volume of liquid was more sensitive because the volume per increment was smaller. No oil layer was present. After the settling was complete and draining of 495 kg of water from the bottom of the tank, analysis of samples and weights indi-

Table 2
Stream masses and compositions

Stream	Meal size	Mass (kg)	Solid (%)	Protein (%)	Ethanol (%)	Oil (%)
Maize meal	2 mm	23	96.0	6.80	–	3.23
	1 mm	21.6	92.9	6.91		3.18
Left in extraction tank	2 mm	14.4	28.3	2.1	–	0.15
	1 mm	3.1	2.0	–		–
Coarse particles	2 mm	4.7	86.9	8.4	–	0.91
	1 mm	7.6	94.1	7.8		1.28
Fine particles	2 mm	7.8	95.2	4.6	–	1.2
	1 mm	4.9	96.7	5.4		1.2
Suspended fine particles	2 mm	0.6	89.1	3.4	–	4.4
	1 mm	0.27	90.1	4.1		18.7
Zein isolate	2 mm	0.13	90.8	73.6	–	7.7
	1 mm	0.14	90.8	73.6		7.7
Extract to settling	2 mm	235.6	0.44	0.17	61.8	0.05
	1 mm	245.5	0.44	0.17	63.0	0.04
Extract liquid	2 mm	255	0.40	0.13	58.9	0.00
	1 mm	181.6	0.43	0.15	65.2	0.04
First centrifugate	2 mm	191	0.38	0.14	58.5	0.03
	1 mm	181.1	0.42	0.16	59.6	0.04
Second centrifugate	2 mm	252	0.71	0.06	55.4	0.02
	1 mm	265.9	0.24	0.05	54.7	0.01

cated that 36 kg of ethanol remained in the 345 kg of liquid left in the settling tank. A plot of the ethanol concentration of the liquid below the interface is shown in Fig. 2. The initial solution of 32 l at 57% ethanol added prior to pumping in the extract is also indicated on the plot for comparison with the top of the liquid pumped from the settling tank.

4. Discussion

We showed previously that corn meal extracts could be separated in small settling tanks of 35-l capacity (Dickey et al., 2003). The particles were removed by circulating water at 1.7 kg of particles/h with a water flow rate 30 times the extract feed rate through a 0.14 m^2 interface in the tank. In this study, extract was fed at a greater velocity of 380 kg/h (31 kg of particles/h) using a larger tank (830-l) with no evidence of particle accumulation below the 0.55 m^2 interface on feed end (one-fourth of total interfacial area) where the highest settling rate (7.6 kg/h) occurred. The water flow at the bottom of this chamber was 20 l/min, about three times the extract feed rate. Restricting the pumped water flow to the bottom of this larger tank prevented particles from accumulating in the tank at this low rate ratio.

The first product liquid was comprised of 217 kg of extract liquid pumped to the settling tank and 32.3 kg at 45% ethanol pumped onto the water before pumping of the extract began. The difference between the product liquid and the liquid fed to the settling tank of 6 kg is the extract liquid entrained with the 12.5 kg of settling particles. The mass ratio, 6/12.5 = 0.5, is an acceptable measure of the effectiveness of the settling. This ratio, using similar 2 mm milled corn, was about 1 for the runs with the smaller settling tank.

The extract was settled for less than 1 h; a longer run would have a lower ratio because the initial mixing before a steady process was established would be lighter for a longer settling period. Initially, the ethanol layer had no particles or oil as the extract (containing oil and particles) was pumped into the settling tank a gradient of particles and oil in the settling layer formed. Dissolved oil that was saturated at extraction temperature separated from solution as the extract cooled and collected at the bottom of the settling extract layer by virtue of its density. This oil layer should impede entrainment of extract liquid. The missing 1.4 kg of oil, the difference between the amount present in the corn meal and that found in the products, is high enough that it must have collected at a location that was not sampled. If the approximately 1.5 l of ‘missing’ oil had been evenly spread over the

full 2.2 m^2 of the extract layer it would have been about 0.7 mm thick. Based on the composition of 6.8% protein of the maize and the usual zein fraction of the protein of about 0.6, the zein available for extraction was 4% of the maize. The maize fraction of the extract was 23/250 (0.092), and had all the zein been put into solution the mass fraction of protein in the extract would have been 0.0037. The extract liquid typically contains only 0.0013 mass fraction of protein indicating that no more than one-third of the zein and one-fifth of the protein fed to the settling tank was transferred to and stayed in the extract liquid after settling.

Estimating the entrainment accurately based on the difference between the extract liquid fed and the recovered fraction is difficult because of the large ratio of the extract liquid to the settling particles, and the small amount of extract used for the first run. This method has been used to estimate entrainment for earlier extractions. A more direct method (measuring the ethanol in the water) was possible using the larger tank because the water could be pumped out of the settling tank (at the end of the settling period) without much interference from the particles in the tank and the ethanol concentration at different levels in the tank measured. This was done in the second run.

The reconstructed ethanol profile obtained from the data of the second run shows that the top 1.4 cm of the final liquid in the settling tank contained 32 l (28 kg) of extract liquid, comprised mostly of the initial solution that was pumped onto the water prior to pumping in the extract. We calculated that 42.8 kg of ethanol that was not part of the product extract liquid were removed from the settling tank. This included 0.92 kg of extract liquid attached to the particles that were pumped out of the tank while pumping extract into the tank (coarse particles) and after the extract pumping was complete (fines).

It will be necessary in the next step of the zein recovery process, if the extract liquid is not reused, to reduce the ethanol concentration to 40% ethanol to precipitate the extracted zein. The 198 kg of recovered extract liquid can be mixed with the 157 l (154 kg) of liquid just below the extract layer, with an average ethanol concentration of 10% to produce a 40% ethanol solution. After the precipitated zein is removed from this mixture, the solution can be distilled to 70% ethanol. Most of the extract liquid in the settling tank must ultimately be recovered from a dilute solution. This does not include solution that can be used to form the zein precipitating mixture (the average 10% ethanol layer), or the top 32 l that is only added at the start of the settling process. Ethanol can be conveniently concentrated (recovered) in the beer still of the associated dry grind plant.

A second measure of extract liquid entrainment that includes the vertical distribution of the extract liquid in the water and the use of the shallow layer of water with the highest extract liquid content is ‘deep entrainment’. Deeply entrained extract liquid ends up in the lower layers of water in the tank and would require distillation from a dilute solution for recovery. For the 1 mm meal, the total entrainment ratio, the mass of extract liquid carried through the interface with the particles/particle mass, is 33.9/12.5 = 2.7. Without the extract liquid in the 157 l (just below the extract layer) in the numerator, the deep entrainment ratio is 9.9/12.5 = 0.8. Applying a similar rationale to the 2 mm meal would lead to a deep entrainment ratio of 0.2 or less. To maintain a steady ethanol concentration at the bottom of the settling tank, some of the aqueous solution containing the deeply entrained extract liquid would have to be pumped out continuously and distilled. This pumping rate would depend on the deep entrainment ratio, and at this rate, the concentration of ethanol in the pumped liquid could be held constant. The mass ratio of the protein in the zein isolate recovered to the protein in the meal extracted and settled was slightly better for the 2 mm meal of 0.085 than for the 1 mm meal of 0.069. This indicated that, despite the larger initial surface area of the finer meal, it did not produce a settled extract with more protein. The yield measured as protein mass in the extract pumped to the settling tank per unit protein extracted was also slightly better for the larger meal size of 0.32–0.28. Complete extraction of zein corresponds to a ratio of about 0.6. About one-fourth of the extracted zein was precipitated as isolate, slightly less than one-fourth was still in the diluted extract liquid after precipitation, and the rest was captured or retained by the settled particles. These results suggest that a maize meal no smaller than 2 mm is preferable for extraction.

5. Conclusions

Particles were settled from maize extracts to water with little dilution of the aqueous ethanol extract liquid. The settled particles were divided into two fractions with different composition by purging the larger particles from the tank with a continuous water flow. The large particles contained more protein than the smaller particles that collected at the bottom of the 830-l settling tank downstream from the extract inlet. Extracted particles can be prevented from accumulating with a much lower ratio of water/extract flow rate than observed in previous studies with a smaller 35-l settling tank. The entrainment of extract liquid, liquid/settled solids of 0.5, was one-half as much as observed for the smaller tank, proba-

bly as a result of the steadier extract/water interface. The finer, 1 mm meal, did not improve the extraction of zein, but did increase the extract liquid entrainment compared with the usual 2 mm screen size. Consequently, the 2 mm meal size is the minimum that should be used with this process.

Acknowledgments

The authors thank Michael F. Dallmer who modified and operated the equipment to produce data used in this report and the mechanical engineering group for designing and fabricating the specialized settling tank.

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